



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

RECEIVED  
SEP 26 2003  
GROUP 1700

In re the application of

Toshiaki Takase et al.

Appln. No.: 09/924,546      Group Art Unit: 1771

Filed: August 9, 2001

For: Battery separator

DECLARATION UNDER 37 C.F.R. 1.132

Honorable Commissioner of Patents and Trademarks  
Washington, D.C. 20231

Sir:

I, Toshiaki TAKASE, citizen of Japan, residing at  
c/o Japan Vilene Company Ltd., 7, Oazakitatone, Sowa-machi,  
Sashima-gun, Ibaraki 306-0213, do sincerely and solemnly  
declare:

THAT I am by profession a chemist having earned a  
Bachelor's degree in analytical chemistry from Faculty of  
Engineering, Department of Applied Chemistry, Niigata  
University in March, 1987;

THAT since April, 1987, I have been an employee of  
Japan Vilene Company Ltd., and since October, 1997, I have  
been engaged in research activities relating to an alkaline  
battery separator;

THAT I am an inventor of the invention of the above-

identified U.S. Patent Application (referred to as "the present invention" hereinbelow) and therefore, am completely familiar with the present invention;

THAT I have reviewed and understand the first Office Action of June 23, 2003, and U.S. Patent No. 6,284,680 (referred to as "the Aikawa et al. Reference" hereinbelow) and Japanese Unexamined Patent Publication (Kokai) No. 11-126595 (referred to as "the JP'595 Reference" hereinbelow); and

THAT in order to show the patentability of the present invention over the Aikawa et al. Reference, in view of the JP'595 Reference, the following experiment was carried out under my direction and supervision.

#### EXPERIMENT

##### (1) Preparation of a separator without high-modulus fibers

The procedure described in Example 1 of the specification of the present application (referred to as "the present specification" hereinbelow) was repeated, except that the high-modulus fibers [i.e., fibers (Young's modulus = 90 cN/dtex) of high-crystalline polypropylene] were not used, to prepare a separator without the high-modulus fibers (referred to as "separator A" hereinbelow).

More particularly, as the islands-in-sea type composite fibers, fibers (fineness = 1.65 dtex, fiber length = 2 mm) having 25 island components of polypropylene in a sea component of poly-L-lactic acid (hereinafter referred to as "PLLA") were prepared by a composite spinning method.

Then, the islands-in-sea type composite fibers were

dipped in a bath (temperature = 80°C) of a 10 mass% aqueous solution of sodium hydroxide for 30 minutes, and the sea component, PLLA, in the islands-in-sea type composite fibers was extracted and removed to obtain polypropylene fine fibers (fiber diameter = 2  $\mu\text{m}$ ,  $\rho/d = 0.083$ , melting point = 172°C, fiber length = 2 mm, density = 0.91 g/cm<sup>3</sup>, sectional shape = circle). The resulting polypropylene fine fibers were drawn and not fibrillated. Each of fibers had substantially same diameter in an axial direction thereof.

Further, as the fusible fibers, sheath-core type composite fibers (Young's modulus = 30 cN/dtex, fineness = 0.8 dtex, fiber length = 5 mm, mass ratio of the core component and the sheath component = 1:1, non-fibrillated, drawn) having a core component (non-fusible component) of polypropylene (melting point = 168°C, density = 0.91 g/cm<sup>3</sup>), and a sheath component (fusible component) of high density polyethylene (melting point = 135°C, density = 0.96 g/cm<sup>3</sup>) was prepared.

Then, 20 mass% of the polypropylene fine fibers and 80 mass% of the sheath-core type composite fibers were mixed, and a fiber web was formed by a wet-laid method (Horizontal Fourdrinier method) from a dispersed slurry.

Then, the fiber web was allowed to stand in a dryer with an internal air circulation at 135°C for 3 minutes, to dry the fiber web and thermally fuse the sheath components (high density polyethylene) of the sheath-core type composite fibers, and thus form a fused nonwoven fabric.

Thereafter, the fused nonwoven fabric was dipped in a solution of fuming sulfuric acid (15%SO<sub>3</sub>) for 2 minutes at 60°C, thoroughly washed with water, and dried to obtain a sulfonated nonwoven fabric having sulfonic acid groups

introduced on the fiber surfaces.

Then, the sulfonated nonwoven fabric was calendered to produce a unilayered-structural separator (i.e., separator A; surface density = 40 g/m<sup>2</sup>, thickness = 0.10 mm, apparent total surface area = 30.9 m<sup>2</sup>, the fibers being substantially two-dimensionally arranged, no bundle of the fine fibers).

(2) Preparation of a separator disclosed in the Aikawa et al. Reference

As a separator disclosed in the Aikawa et al. Reference, a separator (referred to as "separator B" hereinbelow) composed of fibers of which materials are the same as those of fibers forming a nonwoven fabric described in Example 13 of the Aikawa et al. Reference was prepared in accordance with the following procedure.

As the islands-in-sea type composite fibers, fibers (fineness = 1.65 dtex, fiber length = 2 mm) having 25 island components of a mixture (polypropylene = 50 mass%, high-density polyethylene = 50 mass%) of polypropylene (hereinafter referred to as "PP") and high-density polyethylene (hereinafter referred to as "PE") in a sea component of poly-L-lactic acid were prepared by a composite spinning method.

Then, the islands-in-sea type composite fibers were dipped in a bath (temperature = 80°C) of a 10 mass% aqueous solution of sodium hydroxide for 30 minutes, and the sea component, PLLA, in the islands-in-sea type composite fibers was extracted and removed to obtain polypropylene/high-density polyethylene mixed fine fibers (fiber diameter = 2 μm,  $\rho/d = 0.09$ , melting point of PP = 168.7°C, melting point of PE = 129.8°C, fiber length = 2 mm,

sectional shape = circle, PE on the fiber surface = 60% or more). The resulting polypropylene/high-density polyethylene mixed fine fibers were drawn and not fibrillated. Each of fibers had substantially same diameter in an axial direction thereof.

As the fusible fibers, the sheath-core type composite fibers prepared in the above Experiment (1) were used.

Then, 50 mass% of the polypropylene/high-density polyethylene mixed fine fibers and 50 mass% of the sheath-core type composite fibers were mixed, and a fiber web was formed by a wet-laid method (Horizontal Fourdrinier method) from a dispersed slurry.

Then, the fiber web was allowed to stand in a dryer with an internal air circulation at 135°C for 3 minutes, to dry the fiber web and thermally fuse the sheath components (high density polyethylene) of the sheath-core type composite fibers and the PE component of the polypropylene/high-density polyethylene mixed fine fibers, and thus form a fused nonwoven fabric.

Thereafter, a sulfonating treatment and a calendering treatment were carried out in accordance with the procedures described in the above Experiment (1) to produce a unilayered-structural separator (i.e., separator B; surface density = 40 g/m<sup>2</sup>, thickness = 0.10 mm, apparent total surface area = 51.3 m<sup>2</sup>, the fibers being substantially two-dimensionally arranged, no bundle of the fine fibers).

### (3) Evaluation

The uniformity index, the resistance to penetration, and the electrical resistance of separators A and B prepared in the above Experiments (1) and (2) were measured,

in accordance with the procedures described in "Evaluation of Properties" of the present specification.

The results are shown in Table 1, together with the results of the separator prepared in Example 1 of the present specification.

Table 1

	Example 1	Experiment (1) (separator A)	Experiment (2) (separator B)
Surface density (g/m <sup>2</sup> )	40	40	40
Thickness (mm)	0.1	0.1	0.1
Apparent total surface area (m <sup>2</sup> )	29.8	30.9	51.3
Uniformity index	0.08	0.08	0.06
Resistance to penetration (gf)	760	620	670
Electrical resistance ( $\Omega$ )	0.20	0.20	0.19

## RESULTS

As shown in Table 1, the resistance to penetration in the separator prepared in Example 1 of the present specification was superior to that in the separator A (i.e., a separator without high-modulus fibers) or the separator B (i.e., a separator disclosed in the Aikawa et al. Reference and without high-modulus fibers). This result shows that a separator containing high-modulus fibers is superior to that without high-modulus fibers, with respect to the resistance to penetration.

According to the separator containing high-modulus fibers and exhibiting the excellent resistance to penetration, a short circuit caused by a penetration into the separator by a flash generated at the electrode is avoided, and the separator is prevented from being torn by an edge of an electrode, when the separator is wrapped around the electrodes. Therefore, a battery can be reliably assembled. Further, moderate voids are maintained in the separator after wrapping the electrodes, and an electrolyte can be held for a long time. Therefore, a battery having a long lifetime can be assembled.

I, the undersigned declarant, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, or Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Singed this 12th day of September , 2003.

*Toshiaki Takase*

---

Toshiaki TAKASE